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54) Title: FIELD EMITTER FLAT DISPLAY CONTAIN	NING A	GETTER AND PROCESS FOR OBTAIN	ING IT
57) Abstract .			
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"FIELD EMITTER FLAT DISPLAY CONTAINING A GETTER AND PROCESS FOR OBTAINING IT"

The invention relates to a field emitter flat display having an inner vacuum space. The displays of this kind are often referred to as FEDs (Field Emitter Displays) and belong to the wider family of the Flat Panel Displays (FPDs). Said FEDs, as known, also contain, as well as a set of microcathodes, some electric feedthroughs and a plurality of phosphors.

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In detail, a FED contains a plurality of pointed microcathodes (microtips), which emit electrons, and a plurality of grid electrodes, placed at a very short distance from said cathodes, so as to generate a very high electric field; between the cathodes and the phosphors there is a vacuum space, which may be in certain cases some tens to some hundreds of μ m thick. The cathode may also be a diamond emitter. The vacuum degree in the vacuum space is usually kept under 10⁻⁵ mbar with the help of a getter material.

Sometimes the point of the microcathodes, the grid electrodes and the phosphors are aligned on a single flat surface, as described by Henry F. Gray on "Information Display" (3/93, page 11).

The patent document EP-A-0443865 describes a process for preparing a FED wherein a non-conducting substrate, for instance quartz, which supports the microcathodes and possibly the grid electrodes too, in addition to possible auxiliary acceleration-anodes, is coated, in a part thereof free from cathodes and other electrodes, with a thin layer of an evaporable getter alloy based on barium, for instance BaAl₄.

The thus obtained FEDs, however, present some disadvantages; in fact, getters of this kind require, to be operative, an activating heat-treatment (> 800° C) which may be usually carried out by means of radio frequencies, emitted by induction coils outside the FED; in case of an evaporable getter material, the heat-treatment should deposit a film of metal (for instance barium, one of the most commonly used evaporable getters) on well-defined and localized zones of the inner surface of the FED.

As barium is a good electrical conductor, its deposits, especially in a very small space as in the FEDs, may cause short circuits or electric breakdowns of the insulating surfaces; furthermore, said treatment may cause localized thermal shocks so as to seriously endanger the mechanical

resistance of the FEDs.

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Generally, the very small available space hinders the insertion of a getter having enough gas sorption capacity.

Some people, in the past, have proposed to add to the displays an appendix or "tail" C, as shown in Fig. 6, intended to house a getter G without interfering with the thickness of the vacuum space between microtips MT and screen SCH. Such a technique, however, excessively increases the thickness and therefore the volume of the displays.

Said inconvenience - and said appendix - disappear in the displays produced according to the process of the present invention, schematically shown in Fig. 7.

More recently, the application EP-A-572170 suggests to substitute the evaporable getter with other particular kinds of getter, for instance zirconium, which belong to the family of the non-evaporable getters (NEG), preferably present in large amount, such as, for example, microcathodes (microtips).

However, also this suggestion is not free from negative consequences; as a matter of fact, the electronic emission of the sharp point of the microtips, if it is exposed to oxygenated gases, may be changed because of the production of zirconium oxide.

Another disadvantage is due to the difficulties which arise when the microtips are created, usually through a chemical etching of preformed layers; in fact, this technique leaves foreign materials within the microtips, which therefore lose most of their gettering capacity.

Finally, as already mentioned, the oxidation of the microtips, which occurs when these are used as getters, alters the electronic emission characteristics thereof.

It is therefore an object of the present invention to provide a FED, which overcomes at least one of the above mentioned inconveniences of the prior art.

Further objects of the present invention are the elimination of the deposits of getter material or other material on undesired zones inside the FEDs, and the integration of a getter into the very limited space of the FEDs, so as to simultaneously make its manufacture easier.

Other objects will become clear from the following description.

The applicant has succeeded to overcome the above mentioned inconveniences thanks to the present invention.

Said invention, from the widest point of view, consists of a field emitter flat display, having an inner vacuum space wherein there are housed:

- a) a layer of excitable phosphors and a plurality of microcathodes, which emit electrons driven by a high electric field; and
 - b) a plurality of electric feedthroughs and a vacuum stabilizer,

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characterized in that said vacuum stabilizer is essentially formed of a porous supported layer of a non-evaporable getter material, 20 to 180 (preferably 20-150) μ m thick, said layer being housed in a zone essentially free from microcathodes, phosphors and feedthroughs.

In the field of the FEDs there was not, until now, any defined solution of the problems relating to the choice of the getter material and to the method for the manufacture of these FEDs; more precisely, the special features of the FEDs asked pressing and delicate questions about the size, the quality and the easiness of the manufacture, with regard to the production and the conservation of the vacuum necessary for its working.

The displays according to the invention are a successful choice which answers to the above mentioned questions in an extremely satisfying way.

The inner space of the FED according to the invention is preferably defined, as shown in Fig. 7, by two thin plates made of an insulating material, one essentially parallel to the other, hermetically sealed along the perimeter and separated by a high-vacuum space, having a thickness of some tens or hundreds to some thousands of μm . A first plate (SCH) supports the phosphors and the second plate (S) supports the microcathodes, for example made of molybdenum, and possibly also some grid electrodes, for example made of niobium, as well as one or more porous layers of a non-evaporable getter material.

Such layers are then placed between said two thin plates and thus these layers (or thin stripes) are an integral part of the display (FED).

The supported porous layers, present in the displays according to the invention, are based on getter materials having in certain cases a very low activation temperature ($\leq 500^{\circ}$ C and even $\leq 450^{\circ}$ C), which may be applied with different methods on thin metallic and non-metallic substrates, and which may advantageously have, after the application, a possibly long sintering treatment; said treatment strengthens said getter materials, thereby preventing them from losing some particles which are extremely harmful to the above mentioned purposes.

Getter materials particularly suitable to the object are sintered compositions essentially made of:

- A) zirconium and/or titanium and/or thorium and/or the relative hydrides and/or their combinations; and of:
 - B) getter alloys based on zirconium and/or titanium chosen among:
 - i) the Zr-Al alloys, according to USP 3.203.901, and/or Zr-Ni and Zr-Fe alloys according to USP 4.071.335 and USP 4.306.887;
 - ii) the Zr-M1-M2 alloys, according to USP 4.269.624 (where M1 is chosen between V and Nb and where M2 is chosen between Fe and Ni) and the Zr-Ti-Fe alloys, according to USP 4.907.948;
 - iii) the alloys containing zirconium and vanadium and in particular the Zr-V-Fe alloys according to EP-A-93/830411;
 - iv) their combinations.

The compositions known as St 121 and/or St 122, manufactured and commercialized by the applicant, essentially consisting of the two following groups of components:

H) titanium hydride;

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- K) getter alloys chosen among:
- a) Zr-Al alloys according to the aforesaid item B/i), and in particular alloys containing 84% by weight of zirconium (for St 121);
- b) Zr-V or Zr-V-Fe alloys according to the aforesaid item B/iii) (for St 122);
 - c) their combinations,

turned out to be particularly advantageous for the purpose.

The displays according to the invention can be obtained with different methods. According to a particularly advantageous embodiment, said displays are obtained with a process wherein:

- a) said porous layer is obtained by depositing a non-evaporable getter material on a substrate and by sintering the deposited material in a suitable vacuum oven.
- b) the thus obtained supported layer is housed in said inner space together with the other inner components of the display;
- c) said inner space is evacuated by means of a vacuum pump and hermetically sealed during the pumping;

characterized in that the depositing of said getter material on said substrate is carried out by means of electrophoresis or by means of a

manual or mechanical application, preferably spray, of a suspension of said getter material particles in a suspending means.

A mechanical application different from the spray coating may be for example the spreading of said suspension, carried out by one or more panels or by means of a spreading machine with a scraping blade.

With regard to the electrophoretic methods see the previous patents GB-B-2.157.486 and EP-B-0275844, granted to the applicant.

In order to hermetically seal the inner space of the display a frit sealing under vacuum pumping is usually performed, preceded by a high degassing, under vacuum pumping too, from the inner space and from the surrounding walls. The frit sealing and the degassing are carried out at high temperatures, which can be usefully exploited in order to perform the necessary thermal activation of the getter material (without activation a getter cannot perform its functions); all this can be obtained without resorting to anyone of the annoying separate activations, for instance by means of induction coils, which were used in the past. It should be noted, by the way, that this is possible only thanks to the peculiar getter materials selected by the applicant, which have a very low activating temperature.

An even more preferred embodiment of the aforesaid process provides for preparing said porous supported layer of non-evaporable getter material, comprising the following steps:

- a) preparing a suspension of non-evaporable getter material particles in a suspending means;
- b) coating a substrate using said suspension and resorting to the spray coating technique;
 - c) sintering.

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The aforesaid particles are advantageously made of a mixture of:

- H) titanium hydride particles, having an average size essentially comprised between 1 and 10 (preferably 3 to 5) μ m and a surface area of 1 to 8,5 (preferably 7 to 8) m²/g;
- K) getter alloy particles, having an average size essentially comprised between 5 and 15 (preferably 8 to 10) μ m and a surface area of 0,5 to 2,5 m²/g:

wherein said getter alloy is chosen among the Zr-Al alloys, the Zr-V-Fe alloys and their combinations, and wherein the ratio by weight between the H particles and the K particles is 1:10 to 10:1 and preferably 1:1 to 3:1:

By using powders of getter material having the aforesaid particle size and the aforesaid surface area, it is assured a good sorption capacity of the gases emitted during the manufacture of FEDs and during the whole life of the FEDs themselves. Said gases are usually H_2 and gases containing oxygen (such as CO, CO₂, H_2 O, O₂) which are very harmful to the microcathodes points; the sorption capacity in case of CO may reach a value around 0.5×10^{-3} mbar x l/cm².

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One of the dispersing means listed in the aforesaid patent GB-B-2.157.486 or other equivalent means may be used as suspending means.

The porous getter layer may be supported by a metallic substrate, by a conducting non-metallic substrate (for instance silicon) or by an insulating substrate. In case of a metallic substrate, the thickness is usually very thin, for example 5 to 50 μ m; moreover, the substrate may be mono-metallic or multi-metallic, as described in the patent EP-B-0275844.

An example of a metallic substrate is a layer of titanium, molybdenum, zirconium, nickel, chrome-nickel alloys or iron-based alloys, possibly coupled with a layer of aluminum, as described in said patent EP-B-0274844; such a substrate may advantageously be a thin strip, preferably containing holes or slots of any shape, for example round, rectangular, square, polygonal, oval, lobed, elliptical, etc.

Another particular kind of metallic substrate may be one of the non-magnetic alloys, based on iron and manganese, described in EP-A-0577898.

If the substrate is essentially insulating or non-metallic, a suspension of NEG may be directly deposited on such an insulating or non-metallic substrate or a mono-metallic or multi-metallic fixing layer, completely similar to the aforesaid metallic substrates, may be advantageously interposed.

According to an alternative, a suspension of NEG may be separately deposited on a metallic strip and then said strip may be mechanically housed in a micro-groove of the insulating substrate.

In order to perform the spray coating it may be advantageous to use the "multiple cycles" technique. Said technique lies in spraying the affected surface for a very short time, for example few seconds or even less than one second, in breaking off the spraying for a time greater than the previous one, about 10 to 50 seconds, so as to let the volatile liquids evaporate, and then in repeating the spraying step, the evaporating step...and so on, according to the requirements.

The multiple spraying may be advantageously performed with a single nozzle or, alternatively, the repeated use of a single nozzle may be replaced by using a sequence of single-step nozzles, suitably spaced along a support strip in motion; a second alternative provides for using a fixed strip sprayed by means of a sequence of proportioning nozzles in motion.

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The suspensions used within the single cycles may be the same or mutually different; in certain cases it is even possible to spray, in one or more cycles, a suspension of A particles only (or H, for instance titanium hydride) and in a second sequence of one or more cycles a suspension of B particles only (or K, for instance Zr-V of Zr-V-Fe alloys). As an alternative, it is possible to use variable concentrations, for example gradually, of the two kinds of particles.

It is thus possible to advantageously obtain getter layers comprising elementary overlapping layers, having the same or a different composition; those sets of elementary layers, which have on the substrate side one or more elementary layers essentially consisting of titanium particles only, turned out to be very advantageous in view of the adherence to the substrate.

At the end of the spray depositing, the coated substrate is dried by means of a mild air-heating, for example at 70-80° C, and subsequently a vacuum sintering treatment is carried out, at a pressure lower than 10⁻⁵ mbar and at a temperature essentially comprised between 650 and 1200° C.

Here, the term "sintering" means the heating process of a layer of getter material at a temperature and for a time sufficient to give a certain mass transfer among adjacent particles without excessively reducing the surface area. Said mass transfer binds the particles together, thereby increasing the mechanical strength, and enables the adherence of the particles to the support; lower temperatures need longer times. According to a preferred embodiment of the present invention it is chosen a temperature which is the same or slightly higher than the sintering temperature of the H components and slightly lower than the sintering temperature of the K component.

In this description the term "insulating", given to one of the possible substrates, means any material which does not conduct electricity at the working temperature, for example pyroceram, quartz glass, quartz, silica, in general terms refractory metal oxides and in particular alumina.

The invention is now described in greater detail, not in a limiting way, with reference to the attached drawings, wherein:

figures 1 and 2 are micrographies of supported porous layers;

figure 3 is a diagram which reports the results obtained from carbon monoxide sorption tests; 5

figure 4 is a perspective view of a FED insulating substrate ("rear plate") coated by a thin getter stripe having a thickness d, supported on a thin fixing strip, not shown in the drawing, without showing the microcathodes (microtips);

figure 5 is a perspective view of another "rear plate" coated by two stripes instead of one;

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figure 6 is the cross-section view of a FED according to the prior art, provided with a "tail";

figure 7 is the simplified cross-section view of a FED according to the invention.

Reference is made now to Fig. 1, i.e. a 1000x enlarged micrography of a visible surface portion of the layer obtained according to example 1, which clearly shows the high porosity and the good sintering level of the sample.

Fig. 2, i.e. the 1860x enlarged micrography (by backscattering analysis) of a portion of the cross-section of the same layer of example 1 (A-A section in fig. 4), points out, not only the good layer porosity, but also the satisfying distribution uniformity of the sintered mixture components, as well as the good fixing to the Ni-Cr substrate.

Fig. 3 is a graph of the results of the carbon monoxide sorption tests as for the samples obtained according to example 1; for the meaning of the X axis (Q) and the Y axis (G), see the previous international patent application WO 94/02957, with the difference that, in the present case, the sorption of 1 cm2 of exposed surface is concerned. In detail, it should be noted that the sample obtained according to the invention and according to example 1 shows:

- an initial sorption speed of carbon monoxide G₁ equal to approximately 3 l/s x cm²;
- a quantity of sorbed carbon monoxide Q₁ equal to approximately 0,5 x 10⁻³ mbar x l/cm² when speed G is reduced to 0,1 l/s x cm².

The sorption tests were carried out with the following operative

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conditions:

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- sorption temperature: 25°C;
- activation temperature: 500°C (for 10 min.);
- test pressure: 3 x 10⁻⁵ mbar.

Fig. 4 shows a Field Emitter Display, without the fluorescent screen, wherein a quadrangular support is provided with a rectangular stripe of a porous NEG layer, having a thickness d, parallel to one of the sides of the support.

This stripe of porous getter may be thermally activated in an advantageous way by exploiting the same manufacturing process of the FED and in particular the step called frit sealing or the previous degassing step, wherein temperatures around 300-450° C are reached; for details about the term "frit sealing" see the Italian patent application MI93A 002422.

Moreover, the stripe of porous getter may be advantageously connected with one or more electric feedthroughs P, ready for a subsequent further activation, if the latter is needed.

Fig. 5 shows a FED similar to the one in fig. 4, without showing the feedthroughs, provided with two mutually perpendicular stripes, wherein one is longer than the other.

Fig. 6 has been already described in another part of the specification.

Fig. 7 is a cross-section view of a field emitter display (FED) according to the invention, without the "tail", wherein an insulating substrate S and a porous layer of NEG (G) are separated by a metallic fixing strip NS.

The following example is merely given for an explanatory purpose and does not limit in any way the spirit and the scope of the invention.

EXAMPLE

150 g of titanium hydride, having a particle size lower than 60 μ m, were introduced, together with 50 cc of demineralized water, in the steel container of a planetary ball mill.

After the natural evaporation of the water, a powder of titanium hydride having a particle size lower than 20 μ m (average size: 3-5 μ m) was obtained by adjusting the time (about 4 hours) and the milling speed and after the fixing of a suitable number and size combination of the balls in said container. The surface area was 8,35 m²/g.

150 g of St 101 alloy (84% Zr, 16% Al), having a particle size lower than 53 μ m, were milled at the same conditions and with the same parameters used for milling the titanium hydride; a powder consisting of particles having a size lower than 30 μ m (average size: 8-19 μ m) was thus obtained. The surface area was 2,06 m²/g.

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Subsequently, in a plastic bottle, 70 g of said milled titanium hydride were mixed with 30 g of said St 101 alloy, finely milled. These are the typical proportions for forming a composite getter material called St 121. Then, there were added 150 cc of suspending means obtained by mixing 300 cc of isobutyl acetate, 420 cc of isobutyl alcohol and 5,3 g of collodion cotton (nitrocellulose). The bottle was then sealed and mechanically shaken for a time longer than 4 hours.

There has been thus obtained a homogeneous suspension which, if stored for any period, must be shaken again for about two hours before being used.

The suspension was then deposited on the surface of a metallic support by means of a spray system comprising a plastic tank, a pressure-regulated spray needle-valve (model 780S Spray Valve of the EFD company) and a control unit (model Valvemate 7040 by EFD).

For the present example there were used metallic supports made of Ni-Cr, strip-shaped, 0,05 mm thick and 4 mm wide (in other tests sheets 0,02 mm thick have been used).

The valve was supported by a pole so that the spraying nozzle was about 30 cm away from the horizontal surface of the support. The depositing process comprised a sequence of steps (cycles) wherein the valve was opened for a second approximately, thereby letting the suspension flow as tiny droplets, and then closed for a period of 15 seconds approximately, wherein the suspension means could evaporate. In order to accelerate the latter process, the support was kept at about 30° C by means of a heating support plate.

The thickness of the deposit of getter material was proportional to the number of spraying cycles.

The samples coated by a St 121 powder on one face only, were introduced into a vacuum oven, wherein the pressure was reduced to less than 10⁻⁵ mbar; the temperature was then increased up to approximately 450° C, value kept for about 15 minutes.

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Thereafter, the temperature of the oven was increased up to 900° C (sintering temperature) and kept for 30 minutes.

Finally, the system was cooled down to the ambient temperature and the coated supports were extracted from the oven; the deposit of sintered powder was 150 to 180 μm thick along the surface of the metallic support.

Fig. 1 and 2 are the micrographies obtained from the SEM (Scanning Electron Microscopy) analysis of the visible surface of the getter material deposit after being sintered.

Fig. 1, i.e. the 1000x enlarged micrography of a visible surface portion of the getter material layer obtained according to example 1, clearly shows the high porosity and the good sintering level of the sample.

Fig. 2, i.e. the 1860x enlarged micrography (by backscattering analysis) of a portion of the cross-section of the same getter material layer of the example (A-A section in Fig. 4), points out not only the good layer porosity, but also the satisfying uniformity of the distribution of the sintered mixture components, as well as the good fixing to the Ni-Cr substrate.

Fig. 3 (line 1) reports the carbon monoxide sorption tests.

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CLAIMS

- 1. A field emitter flat display, having an inner vacuum space wherein there are housed:
- a) a layer of excitable phosphors and a plurality of microcathodes (MT), which emit electrons driven by a high electric field; and

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- b) a plurality of electric feedthroughs (P) and a vacuum stabilizer (G), characterized in that said vacuum stabilizer (G) is essentially formed of a porous supported layer of a non-evaporable getter material, 20 to 180 μm (preferably 20 to 150 μm) thick, said layer being housed in a zone essentially free from microcathodes, phosphors and feedthroughs.
- 2. A display according to claim 1, characterized in that said inner space is defined by two thin plates (SCH, S) made of an insulating and/or non-metallic conducting material, one essentially parallel to the other, hermetically sealed along the perimeter and separated by a high-vacuum space, having a thickness of some tens or hundreds of μ m, wherein a first plate (SCH) supports said phosphors and the second plate (S) supports said microcathodes (MT) and possibly also a plurality of grid electrodes which generate said high electric field, as well as one or more of said porous layers of non-evaporable getter material (G), as an integral part of the display.
- 3. A display according to claim 1 or 2, characterized in that said getter material (G) is essentially formed of a sintered mixture of particles chosen between the following two groups:
- A) zirconium and/or titanium and/or thorium and/or the relative hydrides and/or their combinations;
 - B) getter alloys based on zirconium and/or titanium chosen among:
 - i) the Zr-Al alloys and/or Zr-Ni and/or Zr-Fe alloys;
 - ii) the Zr-M1-M2 alloys (where M1 is chosen between V and Nb and where M2 is chosen between Fe and Ni) and/or the Zr-Ti-Fe alloys;
 - iii) the alloys containing zirconium and vanadium and in particular the Zr-V-Fe alloys; and
 - iv) their combinations.
- 4. A display according to claim 1, characterized in that the porous 35 layer of non-evaporable getter material is supported on a substrate essentially formed of a mono-metallic or multi-metallic thin strip (NS),

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preferably 5 to 50 µm thick.

- 5. A display according to claim 4, characterized in that said strip (NS) is essentially made of one or more metal, chosen among nickel, titanium, molybdenum, zirconium, chrome-nickel alloys and iron-based alloys.
- 6. A display according to claim 4, characterized in that said strip (NS) contains holes or slots.
- A display according to claim 1, characterized in that the porous layer of non-evaporable getter material (G) is supported on a substrate (S) essentially formed of an insulating material or a non-metallic conducting substrate, preferably separated from said getter material (G) by means of an interposed mono-metallic or multi-metallic fixing layer, completely similar to the strip (NS) according to claim 4.
- 8. A display according to claim 7, characterized in that said insulating substrate has a square, rectangular or at least partially polygonal shape and supports at least a porous layer of said non-evaporable getter material (G), said layer having at least a rectangular surface whose sides are essentially parallel to one of the sides of the substrate.
- 9. A display according to claim 8, characterized in that said substrate (S) has a square or rectangular shape and supports two of said mutually perpendicular layers, having the same or different lengths.
- 10. A display according to claim 3, characterized in that the porous layer of getter material is composed of a sequence of elementary overlapping layers, having the same or a different composition.
- 11. A display according to claim 10, characterized in that one or more elementary layers, among the first ones on the side of the supporting substrate (S), are essentially made of titanium particles only.
- 12. A process for producing a display according to claim 1 or 2, wherein:
- a) said porous layer is obtained by depositing a non-evaporable getter material (G) on a substrate (S;NS) and by sintering the deposited material in a suitable vacuum oven;
- b) the thus obtained supported layer is housed in said inner space together with the other inner components of the display; and
- c) said inner space is evacuated by means of a vacuum pump and hermetically sealed during the pumping,

characterized in that the deposition of said getter material on said substrate is carried out by means of electrophoresis or by means of a manual or mechanical application, preferably spray, of a suspension of said getter material particles in a suspending means.

- 13. A process according to claim 12, characterized in that the porous layer of non-evaporable getter material (G) is thermally activated by connecting the layer to one or more electric feedthrough (P) and by exploiting the electric resistivity of the layer itself.
- 14. A process according to claim 12, characterized in that said inner space is hermetically sealed by means of a frit sealing operation under vacuum pumping, preceded by a degassing operation, also under vacuum pumping, said operations being carried out at high temperatures, which thermally activate the getter material.
- 15. A process according to claim 12, characterized in that the supported porous layer of getter material is obtained by:
- a) preparing a suspension of non-evaporable getter material (G) particles in a suspending means;
- b) coating a supporting substrate by means of said suspension with the spray coating technique; and
 - c) sintering the thus obtained coating.

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- 16. A process according to claim 14, characterized in that said particles are a mixture essentially consisting of:
- H) titanium hydride particles, having an average size essentially comprised between 1 and 15 (preferably 3 to 5) μ m and a surface area of 1 to 8,5 (preferably 7 to 8) m²/g; and
- K) getter alloy particles, having an average size essentially comprised between 5 and 15 (preferably 8 to 10) μ m and a surface area of 0,5 to 2,5 m²/g,

wherein said getter alloy is chosen among the Zr-Al alloys, the Zr-V alloys, the Zr-V-Fe alloys and their combinations, and wherein the ratio by weight between the H particles and the K particles is 1:10 to 10:1 and preferably 1:1 to 3:1.

17. A process according to claim 15, characterized in that the substrate surface is sprayed one or more times (cycles) for a predetermined time and every spraying is followed by a break, which allows a satisfying evaporation of the components of the suspending means, the time of every

- 15 -

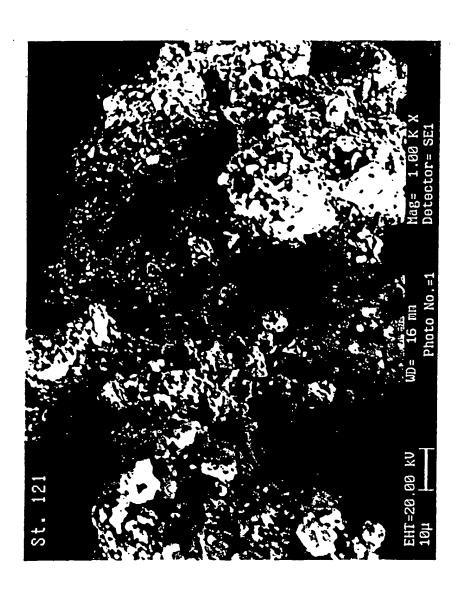
break being longer than the previous spraying time.

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- 18. A process according to claim 17, characterized in that the suspensions used in the single cycles are at least in part mutually different.
- 19. A process according to claim 18, characterized in that the first spraying cycle (or the first 2-3 cycles) are carried out with a suspension containing titanium hydride particles only.

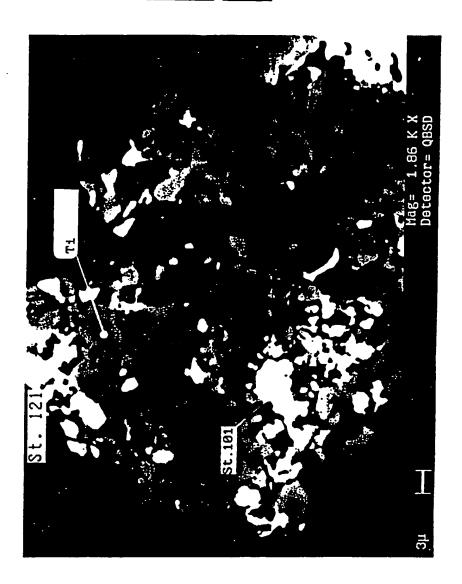
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Fig.1



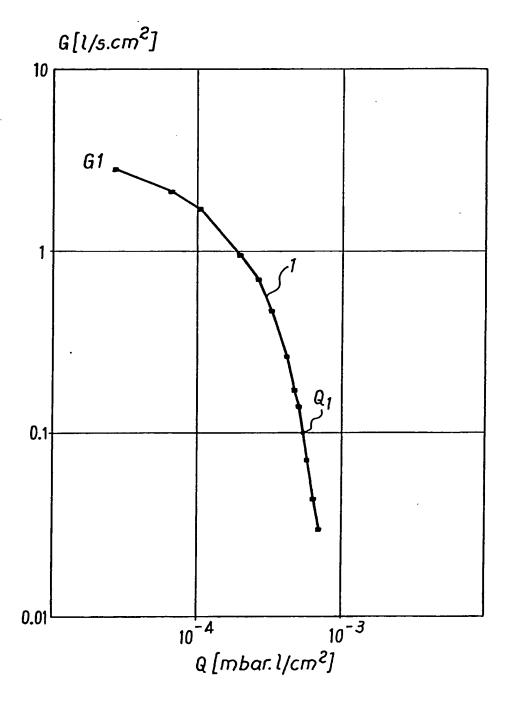
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Fig. 2



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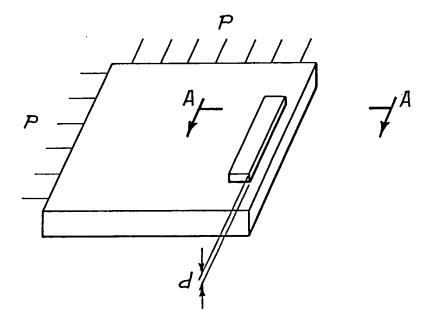
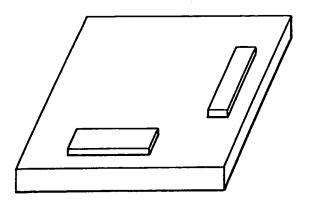
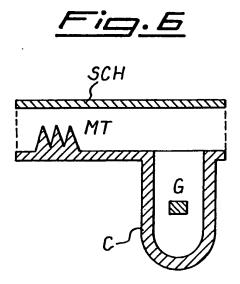
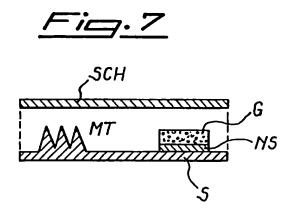


Fig.5







INTERNATIONAL SEARCH REPORT

Inten nal Application No
PCT/IT 95/00031

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A. CLAS IPC 6	H01J31/12 H01J7/18 H01J29	/94	
According	to International Patent Classification (IPC) or to both national cla	assification and IPC	
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IPC 6	documentation searched (classification system followed by classifi H01J	cation symbols)	
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Electronic	data base consulted during the international search (name of data	base and, where practical, sea	rch terms used)
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A	US,A,4 269 624 (FIGINI ALESSANDF 1981 cited in the application see column 2, line 8 - line 60	RO) 26 May	3
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	actual completion of the international search	T	international search report
	June 1995	1 4. 06. 95	·
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